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I also certify that by virtue of an assignment registered under the Patents Act 1977, the application is now proceeding in the name as substituted

I further certify that the attached copy of the request for grant of a Patent (Form 1/77) bears an amendment, effected by this office, following a request by the applicant and agreed to by the Comptroller-General..

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Signed

*Andrew Gervay*

Dated 19 NOV 1999



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INVESTOR IN PEOPLE

GB9822527.9.

By virtue of a direction given under Section 30 of the Patents Act 1977, the application is proceeding in the name of

INTERNATIONAL COATINGS LIMITED,  
Incorporated in the United Kingdom,  
George Street,  
London,  
W1A 2BB,  
United Kingdom

[ADP No. 07558026001]



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Application No: GB 9822527.9  
Claims searched: 1 to 38

Examiner: Miss M M Kelman  
Date of search: 21 January 1999

**Patents Act 1977**  
**Search Report under Section 17**

**Databases searched:**

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.Q): C3B, C3V VCA

Int Cl (Ed.6): C09D 5/03, 5/08, 5/46, 7/12

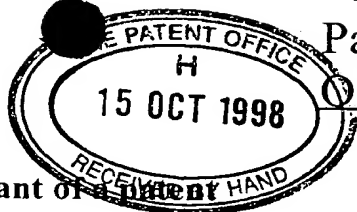
Other: ONLINE: EDOC

**Documents considered to be relevant:**

Category	Identity of document and relevant passage	Relevant to claims
X,Y	GB 2302092 A CIBA-GEIGY see the claims and page 9, lines 13 to 14	X:1,2,3,4, 5,6,7,11,1 7,22,27,28 ,30,34,35, 36 at least Y:2,24,29, 33
Y	GB 1485388 A ALUMINUM COMPANY	2,24,29,33
X	US 5677367 A SAVIN see Examples 5, 11 and 13	1,3,4,27,2 8,30,34,35 ,36 at least
X	US 5338348 A SAVIN see Example 2	1,3,4,27,2 8,30,34,35 ,36 at least

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.





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1/77

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Request for grant of a patent

The Patent Office

Cardiff Road  
Newport  
Gwent NP9 1RH

1. Your reference IP/A19/METALLICS/4289 GB

2. Patent application number  
(The Patent Office will fill in this part) **9822527.9**

3. Full name, address and postcode of the  
or of each applicant (*underline all surnames*) COURTAULDS COATINGS (HOLDINGS)  
LIMITED

Patents ADP number (*if you know it*)

If the applicant is a corporate body,  
give the country/state of its  
incorporation

50 George Street,  
London, W1A 2BB

United Kingdom

SECTION 20 (1977 ACT) APPLICATION FILED 13-6-99.  
05627906002

4. Title of the invention POWDER COATING COMPOSITIONS

5. Name of your agent (*if you have one*) Abel & Imray  
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Kingdom to which all correspondence 303-306 High Holborn  
should be sent (*including the postcode*) London WC1V 7LH

Abel & Imray  
20 Red Lion Street  
LONDON  
WC1R 4PQ

Patents ADP number (*if you know it*) 174001 ✓

6. If you are declaring priority from one Country Priority application Date c  
or more earlier patent applications, give number (day/month/year)  
the country and the date of filing of the  
or of each of these earlier applications  
and (*if you know it*) the or each  
application number

7. If this application is divided or Number of earlier Date of filing  
otherwise derived from an earlier UK application (day/month/year)  
application, give the number and the  
filing date of the earlier application

8. Is a statement of inventorship and of YES  
right to grant of a patent required in  
support of this request? (*Answer 'Yes' if:*  
a) any applicant named in part 3 is not an  
inventor, or  
b) there is an inventor who is not named as an  
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Description 26

Claim(s) 5

Abstract 1

Drawing(s) -

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (*Patents Form 7/77*)

Request for preliminary examination and search (*Patents Form 9/77*)

Request for substantive examination (*Patents Form 10/77*)

Any other documents  
(*please specify*)

11. I/We request the grant of a patent on the basis of this application.

Signature

Date 15.10.98

*A. J. Coulson*

12. Name and daytime telephone number of person to contact in the United Kingdom
- |                   |               |
|-------------------|---------------|
| Mr. A. J. Coulson | 0171 405 0203 |
|                   | 0171 242 9984 |



### POWDER COATING COMPOSITIONS

This invention relates to powder coating compositions, and in particular to powder coating compositions which incorporate pigments providing metallic effects.

Powder coatings form a rapidly growing sector of the coatings market. Powder coatings are solid compositions which are generally applied by an electrostatic spray process in which the powder coating particles are electrostatically charged by the spray gun and the substrate is earthed.

Charging of the powder in the spray gun is effected by means of an applied voltage or by the use of friction (tribo-charging). Conversion of the adherent particles into a continuous coating (including, where appropriate, curing of the applied composition) may be effected by heat treatment and/or by radiant energy, notably infra-red, ultra-violet or electron beam radiation. The powder coating particles which do not adhere to the substrate can be recovered for re-use so that powder coatings are economical in use of ingredients. Also, powder coating compositions are generally free of added solvents, and, in particular, do not use organic solvents and are accordingly non-polluting.

Powder coating compositions generally comprise a solid film-forming resin binder, usually with one or more colouring agents such as pigments, and optionally also contain one or more performance additives. They are usually thermosetting, incorporating, for example, a film-forming polymer and a corresponding curing agent (which may itself be another film-forming polymer), but thermoplastic systems (based, for example, on polyamides) can in principle be used instead. Powder coating compositions are generally prepared by intimately mixing the ingredients (including colouring agents and performance additives) for example in an extruder, at a temperature above the softening point of the film-forming polymer(s) but below a temperature at

which significant oxidation-reaction would occur. The extrudate is usually rolled into a flat sheet and comminuted, for example by grinding, to the desired particle size. Other homogenisation processes also come into consideration, including non-extruder-based processes such as, for example, processes involving mixing using supercritical fluids, especially carbon dioxide.

In coatings formed from compositions incorporating pigments providing metallic effects, generally the pigments are covered by a layer of the film-forming resin binder. Problems arise, however, if there is ingress of moisture and oxygen through the film (which may in some cases be very thin or even non-existent) or if the film is damaged, because the resulting attack on the pigment leads to deterioration of the appearance of the film (blackening, darkening, dark spots, loss of gloss/brilliance, etc.). Current practice frequently involves the use of a clear coating layer applied over the metallic effect layer in order to provide further protection, and/or the use of metallic pigments bearing an inorganic or organic protective coating.

The present invention provides a powder coating composition which comprises a film-forming polymer, a pigment providing a metallic effect, and a corrosion-inhibiting additive.

The pigment providing a metallic effect (hereinafter "metallic pigment") is usually in flake form and may comprise aluminium or an aluminium alloy or another metal or alloy, for example, stainless steel, copper, tin, bronze or brass (gold is generally too expensive) or zinc and may be used to produce various metallic effects including those referred to as "lustre" or "glamour" finishes. Combinations of two or more different metallic pigments may be used.

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~~The metallic pigment is advantageously aluminium or an alloy thereof,~~  
and either a "leafing" or a "non-leafing" system may be used. In a leafing system, the aluminium flakes orient themselves in a continuous layer at or

near the surface of the applied coating film, producing an opaque silver finish.

Non-leafing aluminium pigments, which orient themselves throughout the coating film, provide aesthetics quite unlike leafing aluminium pigments. They are unique in their ability to project "flop", polychromatic and sparkle effects.

- 5 ("Flop" is the ability to change colour when viewed at different angles. This capability is directly related to flake orientation in the film.)

The metallic pigment(s) are usually incorporated in the powder coating composition by dry-blending, and may be incorporated by any available dry-blending method, for example:

- 10 (a) injection at the mill, with the chip and additive(s) fed into the mill simultaneously;
- (b) introduction at the stage of sieving after milling; and
- (c) post-production blending in a "tumbler" or other suitable mixing device.

- Method (b) or (c) will generally be preferred, because the desired
- 15 metallic effect provided, for example, by "leafing" of the pigment may easily be destroyed by strong shear forces. Similar considerations mean that it is not usually recommendable to incorporate the metallic pigment(s) before or during extrusion or other high-shear homogenisation process. The position is different, however, in the case of low-shear homogenisation processes, such
- 20 as processes involving mixing using supercritical fluids, and in such cases some or all of the metallic pigment(s) may expediently be incorporated pre-homogenisation.

- In principle, a proportion of the total content of metallic pigment(s) may be incorporated pre- and/or during the homogenisation process, with the
- 25 remainder being incorporated post-homogenisation.

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The metallic pigment may be incorporated by simple admixture or may be bonded to the powder coating particles, for example, by a mechanofusion technique in which individual flakes of metallic pigment are bonded to the

surface of the powder coating particles. Thus, as soon as the powder begins to melt during stoving the metallic flakes are released and rise to the surface of the powder coating film, thereby producing a good leafing effect and a bright metallic finish. A further advantage of bonding the metallic pigment to the powder particles is the reduced tendency of the powder/pigment combination to segregate during electrostatic application. In a variant of this approach, the corrosion-inhibiting additive may be bonded to the powder particles simultaneously with the metallic pigment.

A range of hammer and other textured metallic finishes can be produced by using, in addition to the metallic pigment, an appropriate hammer or other structure additive.

The metallic pigment may be an uncoated or coated material. Examples of coated materials include pigments coated with silica or another inert inorganic material for greater chemical resistance and durability. Alternatively, the pigment may be coated with a plastics material for similar purposes, for example, an acrylic, PTFE or thermosetting plastics material, or may be carried in a polymer or plasticiser which is compatible with the film-forming binder of the powder coating composition. As a further possibility, the metallic pigment may be coated with a colouring agent such as a metal oxide pigment such as, for example, iron oxide, to provide special colour effects.

In one form of composition according to the invention, the metallic pigment is pre-treated with the corrosion-inhibiting additive (for example, by a solution pre-treatment) so as to form a coating of the additive on the pigment.

An alternative process for introducing the metallic pigment and/or the corrosion-inhibiting additive and/or one or more other additives is the agglomeration technique disclosed in WO 91/18951. The product of that technique in the context of the present invention is a fused or bonded agglomerate of different particulate components comprising a primary film-

forming component and one or more other components (pigments and/or other additives).

The total proportion of metallic pigment(s) incorporated in the powder coating composition by dry blending may be in the range of from 0.1 to 10% by weight (based on the weight of the composition without the metallic pigment(s)), for example, from 0.4 to 8% by weight, preferably from 0.1 to 5% by weight, typically from 1 to 4% by weight.

In the case of incorporation before and/or during homogenisation, especially in the case of low-shear (non-extruder-based) homogenisation processes, the total content of metallic pigment(s) incorporated may be in the range of from 0.1 - 50% by weight, based on the total weight of the composition, preferably at least 5% or 10% by weight, advantageously not exceeding 30% by weight, for example, 5 to 30% or 10 to 30% by weight.

In one form of powder coating composition according to the invention, the corrosion-inhibiting additive comprises at least one silicate material selected from the group consisting of:

- (a) materials obtainable by admixture or reaction of silica or a silicate with a compound of a trivalent metal; and
- (b) naturally occurring or synthetic metal silicates.

Advantageously, the corrosion-inhibiting additive also includes zinc oxide, which may be incorporated in an amount in the range of from 2 to 30% by weight, based on the total weight of the corrosion-inhibiting additive, advantageously at least 5%, 10% or 15% by weight, more especially not exceeding 20% or 25% by weight.

The preferred trivalent metal in embodiment (a) is chromium, iron or aluminium, especially aluminium. Embodiment (b) advantageously uses a silicate of a trivalent metal, and the same preferences apply.

The compound of the trivalent metal may, for example, be a phosphate (preferably a mono- or di-hydrogen phosphate), a fluoride, silicofluoride, chloride, sulphate or alkane carboxylate. Aluminium phosphates or sulphate are preferred.

- 5           The silica in embodiment (b) is advantageously amorphous silica or a precursor thereof.

Examples of naturally occurring metal silicates for use in embodiment (b) are china clay and bentonite.

- The silicate corrosion-inhibiting additive may be surface-modified by  
10   ion exchange. Examples of cations which may be incorporated at the surface of the silicate material by ion exchange include calcium, zinc, cobalt, lead, strontium, lithium, barium and magnesium, especially calcium.

- According to a modification of the invention, the corrosion-inhibiting additive comprises, or is derived from, silica or alumina which has been  
15   surface-modified by ion exchange as described above, preferably in combination with zinc oxide. Further information concerning such surface-modified materials may be found in US 4 687 595 and EP 0 046 057A.  
Examples of calcium-modified silica pigments include those obtainable under the trade name SHIELDEX (Grace) which are based on calcium ion-  
20   exchanged amorphous silica gel. Optionally, such materials may be used in combination with sulphur-containing organic compounds having fused aromatic and heterocyclic rings according to GB 2 302 092A.

- Suitable corrosion-inhibiting additive materials include the non-toxic anti-corrosive pigments which are obtainable under the trade name  
25   ALBRITECT (Albright & Wilson), especially the CC 300 and CC 500 materials. Further information concerning additive materials which may be used may be found in EP O 273 698A, EP O 360 422A, and EP O 486 476A.

The term "silica" as used herein includes materials obtained by pyrogenic and, preferably, wet processes leading to precipitated silicas or silica gels, as well as, in principle, mixed metal-silicon oxides and naturally-occurring materials such as, for example, diatomaceous earth. Silicas for use according to the invention will in general have an amorphous structure. The term "silica" includes silicic acid materials and other precursor materials. Colloidal silica and fumed silica may be mentioned as examples of suitable silica materials.

As an indication of the relative proportions of silica and trivalent metal compound in embodiment (a), the ratio of silicon to trivalent metal atom may be, for example, 1.5 to 30 : 1, advantageously at least 1.5 : 1, 2.5 : 1 or 3.5 : 1, preferably not exceeding 20 : 1, 15 : 1 or 10 : 1. In general, the same silicon: metal ratios are also applicable to embodiment (b), especially in the case where the metal silicate is a silicate of a trivalent metal.

Other classes of corrosion-inhibiting additive which may be used comprise materials based on metal phosphates (especially hydrogen phosphates, ortho-phosphates or polyphosphates) or borates.

Thus, for instance, another corrosion-inhibiting additive which may be used comprises dicalcium phosphate dihydrate, for example, the material "Budite 222" as supplied by Budenheim Chemische Fabrik, or the material "Caliment M" as supplied by Albright & Wilson.

Other corrosion-inhibiting additives which may be used comprise the following:

- (i) Zinc phosphate di-or tetra-hydrate, preferably in the form of spheroidal particles as described in US 5 137 567; or
- (ii) Spheroidal zinc phosphate as a crystalline phase in admixture with an amorphous phase comprising Fe(II) Phosphate and Fe(III) phosphate.

Further information concerning such materials may be found in  
US 5 030 285.

Another corrosion-inhibiting additive of interest comprises zinc  
phosphate (preferably in spheroidal form) modified with zinc molybdate and  
5 rendered organophilic by suitable surface treatment. An example is the  
material Actirox 106 supplied by Microfine Minerals Ltd.

Further corrosion-inhibiting additives which come into consideration  
include the following:

- (iii) alkaline earth hydrogen phosphates in conjunction with alkaline earth  
10 carbonates, optionally also including one or more additives selected  
from fluorosilicates, fluoroborates, alkali and alkaline earth fluorides.  
Further information concerning such materials may be found in  
US 5 126 074;
- (iv) mixtures of magnesium hydrogen phosphate and calcium hydrogen  
15 phosphate;
- (v) polyphosphate hydrates including modified strontium aluminium  
polyphosphate hydrates such as those obtainable under the trade  
names HEUCOPHOS SAPP and HEUCOPHOS SRPP; zinc  
aluminium polyphosphate hydrates such as those obtainable under  
20 the trade name HEUCOPHOS ZAPP; modified zinc calcium aluminium  
polyphosphate silicate hydrates such as those obtainable under the  
trade name HEUCOPHOS ZCPP; and modified calcium aluminium  
polyphosphate silicate hydrates such as those obtainable under the  
trade name HEUCOPHOS CAPP;
- 25 (vi) orthophosphate hydrates including modified zinc calcium strontium  
orthophosphate silicate hydrates such as those obtainable under the  
trade name HEUCOPHOS ZCP; modified zinc aluminium  
orthophosphate hydrates such as those obtainable under the trade



name HEUCOPHOS ZPA; organically treated basic zinc  
orthophosphate hydrates such as those obtainable under the trade  
name HEUCOPHOS ZPO; modified basic zinc molybdenum  
orthophosphate hydrates such as those obtainable under the trade  
5 name HEUCOPHOS ZMP and organic/inorganic modified basic zinc  
orthophosphate or basic zinc phosphate silicate hydrates such as  
those obtainable under the trade names HEUCOPHOS ZPZ and ZBZ,  
respectively;

(vii) phosphosilicates such as, for example, calcium strontium zinc  
10 phosphosilicate, calcium phosphosilicate, and zinc aluminium  
phosphosilicate;

(viii) zinc aluminium phosphate;

(ix) barium metaborate monohydrate;

(x) zinc salts of organic nitro compounds; and

15 (xi) zinc oxide/cinnamic acid combinations.

For safety and environmental reasons, the use of lead compounds,  
chromates or other toxic materials is not recommended.

Corrosion-inhibiting additives used according to the invention are  
advantageously incorporated by intimate mixing with the other constituent(s)  
20 of the composition, excluding dry-blended additives, before the extrusion or  
other homogenisation process and/or may be incorporated during the  
process. Additionally or alternatively, the additive(s) of the invention may be  
incorporated in the powder coating composition by any available dry-blending  
method, for example:

25 (a) injection at the mill, with the chip and additive(s) fed into the mill  
simultaneously;

(b) introduction at the stage of sieving after milling; and

(c) post-production blending in a "tumbler" or other suitable mixing device.

The individual components of a corrosion-inhibiting additive used according to the invention may be incorporated separately or pre-mixed prior to incorporation (whether pre-, during or post-extrusion or other homogenisation process), or, in the case of embodiment (a), the components may be pre-reacted before incorporation. By way of example, such pre-reaction may be carried out in aqueous solution or dispersion, suitably under acid conditions generated, for example, by incorporation of phosphoric acid, and the resulting precipitate may be washed and thereafter dried at elevated temperature (for example, 100° to 350°C.).

In principle, a proportion or component of the corrosion-inhibiting additive may be incorporated pre- and/or during the homogenisation process, with the remaining proportion or component being incorporated post-homogenisation.

The proportion of corrosion-inhibiting additive(s) incorporated in a powder coating composition of the invention pre- or during homogenisation may in general be in the range of from 0.5 to 50% by weight, based on the total weight of the composition, for example at least 1%, 5% or 10% by weight, for example not exceeding 30% or 40% by weight. Ranges which may be mentioned include from 5 to 20% or 5 to 15% by weight, advantageously from 8 to 12% by weight. The proportion of additive incorporated by dry-blending will in general be significantly less, for example, no more than 5% by weight, although higher proportions (say, up to 7.5% or 10% by weight) may be used in some cases.

The particle size of the or each corrosion-inhibiting additive or component thereof may in general be up to 25 microns, and is preferably no more than 10 microns, especially in the case of thin-film applications. The preferred minimum particle size is 0.1 microns and a range of 2.5 to 7.5 microns may be mentioned as suitable.

The particle size of any zinc oxide included in the corrosion-inhibiting additive is advantageously in the range of from 0.1 to 10 microns.

The particle size distribution of the powder coating composition may be in the range of from 0 to 120 microns with a mean particle size in the range  
5 of from 15 to 75 microns, preferably at least 20 or 25 microns, advantageously not exceeding 50 microns, more especially 20 to 45 microns.

In the case of relatively fine size distributions, especially where relatively thin applied films are required, for example, the powder coating composition may be one in which one or more of the following criteria is  
10 satisfied:

- a) 95-100% by volume < 50  $\mu\text{m}$
- b) 90-100% by volume < 40  $\mu\text{m}$
- c) 45-100% by volume < 20  $\mu\text{m}$
- d) 5-100% by volume < 10  $\mu\text{m}$  preferably 10-70% by volume < 10  
15  $\mu\text{m}$
- e) 1-80% by volume < 5  $\mu\text{m}$  preferably 3-40% by volume < 5  $\mu\text{m}$
- f)  $d(v)_{50}$  in the range 1.3-32  $\mu\text{m}$  preferably 8-24  $\mu\text{m}$

A powder coating composition according to the invention may contain a single film-forming powder component comprising one or more film-forming  
20 resins or may comprise a mixture of two or more such components.

The film-forming resin (polymer) acts as a binder, having the capability of wetting pigments and providing cohesive strength between pigment particles and of wetting or binding to the substrate, and melts and flows in the curing/stoving process after application to the substrate to form a  
25 homogeneous film.

The or each powder coating component of a composition of the invention will in general be a thermosetting system, although thermoplastic

systems (based, for example, on polyamides) can in principle be used instead.

When a thermosetting resin is used, the solid polymeric binder system generally includes a solid curing agent for the thermosetting resin;

5 alternatively two co-reactive film-forming thermosetting resins may be used.

The film-forming polymer used in the manufacture of the or each component of a thermosetting powder coating composition according to the invention may be one or more selected from carboxy-functional polyester resins, hydroxy-functional polyester resins, epoxy resins, and functional

10 acrylic resins.

A powder coating component of the composition can, for example, be based on a solid polymeric binder system comprising a carboxy-functional polyester film-forming resin used with a polyepoxide curing agent. Such carboxy-functional polyester systems are currently the most widely used

15 powder coatings materials. The polyester generally has an acid value in the range 10-100, a number average molecular weight  $M_n$  of 1,500 to 10,000 and a glass transition temperature  $T_g$  of from 30°C to 85°C, preferably at least 40°C. The poly-epoxide can, for example, be a low molecular weight epoxy compound such as triglycidyl isocyanurate (TGIC), a compound such as  
20 diglycidyl terephthalate condensed glycidyl ether of bisphenol A or a light-stable epoxy resin. Such a carboxy-functional polyester film-forming resin can alternatively be used with a bis(beta-hydroxyalkylamide) curing agent such as tetrakis(2-hydroxyethyl) adipamide.

Alternatively, a hydroxy-functional polyester can be used with a  
25 blocked isocyanate-functional curing agent or an amine-formaldehyde

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condensate such as, for example, a melamine resin, a urea-formaldehyde resin, or a glycol ural formaldehyde resin, for example the material "Powderlink 1174" supplied by the Cyanamid Company, or hexahydroxymethyl melamine.

A blocked isocyanate curing agent for a hydroxy-functional polyester may, for example, be internally blocked, such as the uret dione type, or may be of the caprolactam-blocked type, for example isopherone diisocyanate.

As a further possibility, an epoxy resin can be used with an amine-  
5 functional curing agent such as, for example, dicyandiamide. Instead of an amine-functional curing agent for an epoxy resin, a phenolic material may be used, preferably a material formed by reaction of epichlorohydrin with an excess of bisphenol A (that is to say, a polyphenol made by adducting bisphenol A and an epoxy resin). A functional acrylic resin, for example a  
10 carboxy-, hydroxy- or epoxy-functional resin can be used with an appropriate curing agent.

Mixtures of film-forming polymers can be used, for example a carboxy-functional polyester can be used with a carboxy-functional acrylic resin and a curing agent such as a bis(beta-hydroxyalkylamide) which serves to cure both  
15 polymers. As further possibilities, for mixed binder systems, a carboxy-, hydroxy- or epoxy-functional acrylic resin may be used with an epoxy resin or a polyester resin (carboxy- or hydroxy-functional). Such resin combinations may be selected so as to be co-curing, for example a carboxy-functional acrylic resin co-cured with an epoxy resin, or a carboxy-functional polyester  
20 co-cured with a glycidyl-functional acrylic resin. More usually, however, such mixed binder systems are formulated so as to be cured with a single curing agent (for example, use of a blocked isocyanate to cure a hydroxy-functional acrylic resin and a hydroxy-functional polyester). Another preferred  
25 formulation involves the use of a different curing agent for each binder of a mixture of two polymeric binders (for example, an amine-cured epoxy resin used in conjunction with a blocked isocyanate-cured hydroxy-functional acrylic resin).

Other film-forming polymers which may be mentioned include functional fluoropolymers, functional fluorochloropolymers and functional fluoroacrylic polymers, each of which may be hydroxy-functional or carboxy-functional, and may be used as the sole film-forming polymer or in conjunction  
5 with one or more functional acrylic, polyester and/or epoxy resins, with appropriate curing agents for the functional polymers.

Other curing agents which may be mentioned include epoxy phenol novolacs and epoxy cresol novolacs; isocyanate curing agents blocked with oximes, such as isophorone diisocyanate blocked with methyl ethyl ketoxime,  
10 tetramethylene xylene diisocyanate blocked with acetone oxime, and Desmodur W (dicyclohexylmethane diisocyanate curing agent) blocked with methyl ethyl ketoxime; light-stable epoxy resins such as "Santolink LSE 120" supplied by Monsanto; and alicyclic poly-epoxides such as "EHPE-3150" supplied by Daicel.

15 Certain alloys, especially certain aluminium alloys (including alloys used in aerospace applications) undergo metallurgical changes at temperatures of the order of 150°C and above. Accordingly, an important embodiment of the invention comprises so-called "low-bake" compositions formulated so as to be convertible into continuous coating form (with curing as  
20 appropriate) at temperatures not exceeding 150°C, advantageously not exceeding 140°C and preferably not exceeding 130°C.

A powder coating composition for use according to the invention may be free from added colouring agents, but usually contains one or more such agents (pigments or dyes) and can contain one or more performance  
25 additives such as a flow-promoting agent, a plasticiser, a stabiliser, for example a stabiliser against UV degradation, an anti-gassing agent, such as benzoin, a filler, or two or more such additives may be present in the coating composition. Examples of pigments which can be used are inorganic

pigments such as titanium dioxide, red and yellow iron oxides, chrome pigments and carbon black and organic pigments such as, for example, phthalocyanine, azo, anthraquinone, thioindigo, isodibenzanthrone, triphendioxane and quinacridone pigments, vat dye pigments and lakes of  
5 acid, basic and mordant dyestuffs. Dyes can be used instead of or as well as pigments.

A pigment content of  $\leq 40\%$  by weight of the total composition (disregarding dry blend additives) may be used. Usually a pigment content of 25-30% is used, although in the case of dark colours opacity can be obtained  
10 with  $\leq 10\%$  by weight of pigment. Where appropriate, a filler may be used to assist opacity, whilst minimising costs.

In general, colouring agents and performance additives as described above will not be incorporated by dry blending, but will be incorporated before and/or during the extrusion or other homogenisation process.

15 A powder coating composition according to the invention may in principle be applied to a substrate by any of the processes of powder coating technology, for example, by electrostatic spray coating (corona-charging or tribo-charging); or by fluidised-bed or electrostatic fluidised-bed processes.

After application of the powder coating composition to a substrate,  
20 conversion of the resulting adherent particles into a continuous coating (including, where appropriate, curing of the applied composition) may be effected by heat treatment and/or by radiant energy, notably infra-red, ultra-violet or electron beam radiation.

The invention is applicable over a wide range of applied film  
25 thicknesses, typically from thin films of, for example, 30 microns or less up to films of 50, 100, 150 or 200 microns. A typical minimum film thickness is 5 microns, and ranges of 15 to 25 microns and 15 to 40 microns are important.

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A powder coating composition of the invention may incorporate, by dry-blending, one or more fluidity-assisting additives, for example, those disclosed in WO 94/11446, and especially the preferred additive combination disclosed in that Specification, comprising aluminium oxide and aluminium hydroxide, typically in proportions in the range of from 1 : 99 to 99 : 1 by weight, advantageously from 10 : 90 to 90 : 10, preferably from 30 : 70 to 70 : 30, for example, from 45 : 55 to 55 : 45. The use of such additive combinations is especially applicable in the case of relatively fine size distributions as described above, where relatively thin applied films are required. Other dry-blended additives which may be mentioned include aluminium oxide and silica (which may be a wax-coated silica) either singly or in combination with each other or in other additive combinations, for example, including aluminium hydroxide.

A preferred combination comprises wax-coated silica, aluminium oxide and aluminium hydroxide. In the case of additives comprising or consisting of aluminium oxide and/or aluminium hydroxide, preference may be given to  $\gamma$ -structural types.

The amount of fluidity-assisting additive(s) incorporated by dry blending may be in the range of from, for example, 0.05 or 0.1 to 5% by weight, based on the total weight of the composition without the additive(s), advantageously from 0.1 to 3% by weight, more especially 0.1 to 2% by weight, preferably at least 0.2% by weight, especially from 0.2 to 1.5% by weight, more especially 0.3 to 1% by weight.

The total content of non-film-forming additive(s) incorporated by dry blending will in general be no more than 10% by weight, based on the total weight of the composition without the additive(s).

The substrate may, for example, comprise aluminium, iron, tin or zinc. Alternatively, the substrate may comprise alloys of one or more of those



metals with themselves or with other metals such as, for example, copper, nickel and/or magnesium. The metal or alloy may be in the form of a layer on a substrate formed of another material, which may itself be another metal or alloy.

5           The substrate may instead comprise a non-metallic material such as a plastics material, wood, a wood-based product, glass, glass fibre or a composite, ceramic or textile material.

Advantageously, a metal substrate is chemically or mechanically cleaned prior to application of the composition, and is preferably subjected to  
10   chemical pre-treatment, for example with iron phosphate, zinc phosphate or chromate. Substrates other than metallic are in general pre-heated prior to application or, in the case of electrostatic spray application, are pre-treated with a material that will aid such application.

In a typical composition according to the invention, the proportion of  
15   film-forming polymer (and curing agent where appropriate) may be in the range of from 25 to 99.5% by weight, preferably from 40 to 98% by weight, with a proportion in the range of from 40 to 98% by weight, preferably from 50 to 90 or 95% by weight, being representative of compositions including colouring agents.

20           The following Examples illustrate the invention:

The stabilising (corrosion-inhibiting) additives used in the Examples were as follows:

Albritect CC500 :	(Albright & Wilson) a blend of an insoluble aluminium silicate with zinc oxide.
25   Actirox 106:	zinc phosphate (small, spherical particles)
	modified with zinc molybdate and rendered organophilic (Microfine Minerals Ltd.).

Budit 222:

dicalcium phosphate dihydrate (Budenheim  
Chemische Fabrik).

Example 1

A grey polyester powder coating composition was prepared according  
5 to the following formulation (parts by weight):

	Carboxy-functional polyester resin	700
	Primid XL-552	30
	Polyvinyl butyral flow agent	14
	Benzoin	4
10	Amine-modified wax lubricant	8
	Anti-oxidant	2
	Rutile titanium dioxide	100
	Ultramarine blue 54	1.88
	Iron oxide black	0.56
15	Iron oxide red	0.26
	Calcium carbonate extender	<u>120</u>
		980.7

The ingredients were dry-mixed in a blender and fed into a twin-screw  
extruder operating at a temperature of 130°C. The extrudate was rolled flat  
20 on a chilled plate and broken into chip form (c. 1cm mesh). 0.1% by weight of  
Acematt TS100, a silica flattening agent, was added to the chip as a dry-flow  
additive to aid milling. The resulting composition in chip form was ground in  
an impact mill to produce a powder coating composition having a particle size  
distribution within the following range (determined using a Malvern

25 Mastersizer X):

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$$d(v)_{99} = 103-108\mu\text{m}$$

$$d(v)_{50} = 38-42\mu\text{m}$$

$$7-8\% < 10\mu\text{m}$$

1.4% by weight, calculated on the base powder without the metallic pigment, of leafing aluminium powder Special PC100 (from ECKART) was added to the powder coating composition prepared as described above. The aluminium pigment was dry-blended with the powder by shaking until it was  
5 evenly distributed throughout the powder, creating a metallic powder coating composition A.

A was divided into sub-samples. Powder coating compositions according to the invention were produced from several of these sub-samples by dry-blending, again by shaking, stabilising additives into the powder. The  
10 compositions of the additive-containing samples are given below (all quantities are percentages by weight, based on the metallic powder without the additive):

A1	composition A + 1% Albriect CC500
A2	A + 2% Albriect CC500
A3	A + 3% Albriect CC500
A4	A + 2% Actirox 106
A5	A + 3% Actirox 106
A6	A + 2% Budit 222
A7	A + 3% Budit 222

The compositions listed above were applied by electrostatic spray onto aluminium test panels. The powders were stoved at 200° for 8 minutes  
15 to produce cured metallic-effect films of thicknesses 50-80µm.

Panels of the above cured coatings were placed in a humidity cabinet and subjected to condensing humidity under constant conditions, i.e., 100% RH and  $(40 \pm 3)^{\circ}\text{C}$ , according to DIN 50017 (KK). More than one panel was  

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exposed for some samples, because exposure conditions can vary in different  
20 areas of the humidity cabinet. At intervals the panels were inspected and the degree of attack rated according to the following scale:

- 0= no effect
- 1 = isolated dark or discoloured spots
- 2 = numerous spots / slight loss of brilliance
- 3 = pronounced spots / loss of brilliance
- 4 = predominantly spotted / marked loss of brilliance
- 5 = complete destruction

The results after various exposure durations are shown in the following table:

Sample	Rating (duration)
A	5 (50hrs)
A1	4-5 (68 hrs)
A2	3 (68 hrs)
A3	1 (120 hrs)
A4	0-1 (120 hrs)
A5	0-1 (120 hrs)
A6	0 (120 hrs)
A7	0 (120 hrs)

As can be seen, each of the additives improved the resistance of the metallic pigment to discolouration and loss of Brilliance to some extent. Budit 222 and Actirox 106 produced marked improvements, with those films containing Budit 222 showing no signs of moisture and oxygen attack after 120 hours' exposure. A lesser degree of resistance was imparted by Albritect CC500. The latter, however, shows an increase in efficacy with rising levels of additive incorporation.

Example 2

A grey polyester powder coating composition was prepared according to the following formulation (parts by weight):

	Carboxy-functional polyester resin	800
5	Primid XL-552	30
	Polyvinyl butyral flow agent	16
	Benzoin	4
	Amine-modified wax lubricant	6
	Hindered amine light stabiliser	4
10	Rutile titanium dioxide	75
	Iron oxide black	8
	Iron oxide red	0.46
	Iron oxide yellow	0.84
	Blanc Fixe extender	<u>75</u>
15		1019.3

The ingredients were dry-mixed in a blender and fed into a twin-screw extruder operating at a temperature of 130°C. The extrudate was rolled flat on a chilled plate and broken into chip form (c. 1cm mesh). The resulting composition in chip form was ground in an impact mill to produce powder  
20 coating compositions having a particle size distribution within the range described in Example 1.

3.5% by weight, calculated on the base powder without the metallic pigment, of non-leafing aluminium pigment Aluminiumstabil 7654 (Benda-Lutz) was added to the powder coating composition prepared as described  
25 above. The aluminium pigment was bonded to the powder by a  

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mechanofusion technique, creating metallic powder coating composition B.

B was divided into sub-samples. Powder coating compositions according to the invention were produced from several of these sub-samples

by dry-blending, by shaking, stabilising additives into the powder. The compositions of the additive-containing samples are given below (all quantities are percentages by weight, based on the metallic powder without the additive):

5

- B1 composition B + 0.5% Albritect CC500
- B2 B + 1% Albritect CC500
- B3 B + 2% Albritect CC500
- B4 B + 3% Budit 222

These compositions were applied by electrostatic spray onto aluminium test panels. The powders were stoved at 200°C for 8 minutes to produce cured metallic-effect films of thicknesses 50-80µm.

10 Panels of the above cured coatings were placed in a humidity cabinet and subjected to condensing humidity as described in Example 1. At intervals the panels were removed and the degree of attack rated according to the scale detailed in Example 1.

The results after various exposure durations are shown in the following table:

Sample	Rating (duration)
B	3 (48 hrs); 2 (120 hrs); 5 (144 hrs)
B1	2-3 (120 hrs)
B2	0 (68 hrs)
B3	0 (68 hrs)
B4	0 (240 hrs); 1-2 (720 hrs)

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As can be seen, Albritect CC500 again gave an increase in protection with rising level of incorporation, and was more effective at levels of 1% and greater. The improvement afforded by the addition of Albritect CC500 was

greater than that observed with the dry-blended leafing pigment in Example 1. Budit 222 again gave a superior degree of protection, resisting severe attack for several weeks in condensing humidity.

Example 3

- 5 A clear polyester powder coating composition was prepared according to the following formulation (parts by weight):

	Carboxy-functional polyester resin	1000
	Primid XL-552	39
	Hydroxylated polyester flow aid	50
10	Benzoin	4
	Grilonit V68/31	6
	Hindered amine light stabiliser	2
	Anti-oxidant	8
	Pigment violet 11	<u>1.6</u>
15		1110.6

- The ingredients were dry-mixed in a blender and fed into a twin-screw extruder operating at a temperature of 130°C. The extrudate was rolled flat on a chilled plate and broken into chip form (c. 1cm mesh). The resulting composition in chip form was ground in an impact mill to produce powder
- 20 coating compositions having a particle size distribution within the range described in Example 1.

- 1.1% by weight, calculated on the base powder without the metallic pigment, of leafing aluminium powder 1081 from (Benda-Lutz) added to the powder coating composition prepared as described above. The aluminium
- 25 pigment was bonded to the powder by a mechanofusion technique, creating
- 
- metallic powder coating composition C.

C was divided into sub-samples. Powder coating compositions according to the invention were produced from several of the sub-samples by

dry-blending, by shaking, stabilising additives into the powder. The compositions of the additive-containing samples are given below (all quantities are percentages by weight, based on the metallic powder without the additive):

C1 composition C + 1% Albritect CC500

C2 C + 2% Albritect CC500

C3 C + 3% Budit 222

5        These compositions were applied by electrostatic spray onto aluminium test panels. The powders were stoved at 200°C for 8 minutes to produce cured metallic-effect films of thicknesses 50-80µm.

      Panels of the above cured coatings were placed in a humidity cabinet and subjected to condensing humidity as described in Example 1. At intervals  
10    the panels were removed and the degree of attack rated according to the scale detailed in Example 1.

      The results after various exposure durations are shown in the following table:

Sample	Rating (duration)
C	4 (48 hrs); 4-5 (120 hrs)
C1	4 (68 hrs)
C2	3 (68 hrs)
C3	1 (330 hrs)

15        As can be seen, Albritect CC500 gave a small increase in protection with rising level of incorporation. Budit 222 however produced a markedly superior degree of protection, showing only slight attack after 330 hours' exposure to humidity and oxygen.



Example 4 (additives incorporated into base powder)

Epoxy-polyester hybrid powder coating compositions D-H were prepared according to the formulations below (parts by weight). D is a control reference, (not in accordance with the invention) and E-H are compositions according to the invention incorporating stabilising additives.

	D	E	F	G	H
Uralac 5173 polyester resin	500	500	500	500	500
Aralidite GT6063 epoxy resin	225	225	225	225	225
Polyvinyl butyral flow aid	16	16	16	16	16
10 Carnauba wax	6	6	6	6	6
Benzoin	4	4	4	4	4
Calcium carbonate extender	300	-	-	-	-
Albritect CC500	-	300	-	-	-
Actirox 106	-	-	300	-	200
15 Budit 222	-	-	-	300	100
	1051	1051	1051	1051	1051

The ingredients were dry-mixed in a blender and fed into a twin-screw extruder operating at a temperature of 120°C. The extrudate was rolled flat on a chilled plate and broken into chip form (c. 1cm mesh). 0.2% by weight of alumina was added to the chip as a dry flow additive to aid milling. The resulting compositions in chip form were ground in an impact mill to produce powder coating compositions having a particle size distribution within the range described in Example 1.

1.4% by weight, calculated on the base powder without the metallic pigment, of leafing aluminium powder Special PC100 was added to each powder coating composition D-H. The aluminium pigment was dry-blended with each powder by shaking until it was evenly distributed throughout the powder.

The metallic powder coating compositions thus prepared were applied by electrostatic spray onto aluminium test panels. The powders were stoved at 200°C for 8 minutes to produce cured metallic-effect films of thicknesses 50-80µm.

- 5            Panels of the above cured coatings were placed in a humidity cabinet and subjected to condensing humidity as described in Example 1. At intervals the panels were removed and the degree of attack rated according to the scale detailed in Example 1.

             The results after various exposure durations are shown in the  
10    following table:

Powder composition (containing A1 PC100)	Rating (duration)
D	4 (24 hrs)
E	4 (120 hrs)
F	0 (120 hrs)
G	0 (120 hrs)
H	0 (120 hrs)

As can be seen, an improvement in the resistance to degradation of the aluminium pigment is also afforded by the addition of stabilising additives to the base powder prior to extrusion, instead of post-blending into finished  
15    metallic powder. Incorporation of Albritect CC500 into the base powder gives a small improvement, extending the time taken to degrade to a given level. However the addition of Budit 222, Actirox 106 or a mixture of these two to the base powder yields a significantly larger improvement, showing no effect  
whatsoever of moisture and oxygen attack after 120 hours.

CLAIMS

1. A powder coating composition which comprises a film-forming polymer, a pigment providing a metallic effect, and a corrosion-inhibiting additive.
- 5 2. A powder coating composition as claimed in claim 1, wherein the metallic pigment is in flake form.
3. A powder coating composition as claimed in claim 1 or claim 2, wherein the metallic pigment comprises aluminium or an aluminium alloy, stainless steel, copper, tin, bronze or brass or zinc.
- 10 4. A powder coating composition as claimed in any one of claims 1 to 3, wherein the metallic pigment is incorporated in the composition by dry blending, preferably after milling.
5. A powder coating composition as claimed in claim 4, wherein the total proportion of metallic pigment(s) incorporated in the composition by dry  
15 blending is in the range of from 0.1 to 10% by weight, based on the weight of the composition without the metallic pigment(s), for example from 0.4 to 8% by weight, preferably from 0.1 to 5% by weight or from 1 to 4% by weight.
6. A powder coating composition as claimed in any one of claims 1 to 3, wherein the metallic pigment is incorporated in the composition before and/or  
20 during homogenisation, especially in the case of a low-shear homogenisation process, and the content of metallic pigment(s) incorporated is in the range of from 0.1 to 50% by weight, based on the total weight of the composition, preferably at least 5% or 10% by weight, advantageously not exceeding 30% by weight.
- 25 7. A powder coating composition as claimed in any one of claims 1 to 6,  
wherein the a corrosion-inhibiting additive comprises at least one silicate material selected from the group consisting of:

(a) materials obtainable by admixture or reaction of silica or a silicate with a compound of a trivalent metal; and

(b) naturally occurring or synthetic metal silicates.

8. A powder coating composition as claimed in claim 7, wherein the corrosion-inhibiting additive also includes zinc oxide.

9. A powder coating composition as claimed in claim 8, wherein the corrosion-inhibiting additive includes an amount of zinc oxide in the range of from 2 to 30% by weight, based on the total weight of the corrosion-inhibiting additive, advantageously at least 5%, 10% or 15% by weight, more especially not exceeding 20% or 25% by weight.

10. A powder coating composition as claimed in any one of claims 7 to 9, wherein the trivalent metal in embodiment (a) is chromium, iron or aluminium, especially aluminium.

11. A powder coating composition as claimed in any one of claims 7 to 9, wherein the silicate in embodiment (b) is a silicate of a trivalent metal, especially chromium, iron or aluminium, more especially aluminium.

12. A powder coating composition as claimed in any one of claims 7 to 10 in which the compound of a trivalent metal in embodiment (a) is a phosphate, fluoride, silicofluoride, chloride, sulphate or alkane carboxylate.

13. A powder coating composition as claimed in any one of claims 7 to 10 or 12, wherein the silica in embodiment (a) is amorphous silica or a precursor thereof.

14. A powder coating composition as claimed in any one of claims 7 to 13 wherein the corrosion-inhibiting additive, or a silica or silicate used in embodiment (a), is surface-modified by ion exchange.

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15. A powder coating composition as claimed in claim 14, wherein the ions involved in the surface modification are selected from calcium, zinc, cobalt, lead, strontium, lithium, barium and magnesium, especially calcium.

16. A powder coating composition as claimed in claim 14 or claim 15, modified in that the corrosion-inhibiting additive comprises, or is derived from, silica or alumina which has been surface-modified as defined in those claims, preferably in combination with zinc oxide.

5 17. A powder coating composition as claimed in any one of claims 7 to 16, wherein the ratio of silicon to metal atom is in the range of from 0.2 to 30 : 1, advantageously at least 0.5 : 1, 1.5 : 1, 2.5 : 1 or 3.5 : 1, preferably not exceeding 20 : 1, 15 : 1 or 10 : 1.

18. A powder coating composition as claimed in any one of claims 1 to 6,  
10 wherein the corrosion-inhibiting additive comprises a metal phosphate (especially a hydrogen phosphate, ortho-phosphate or polyphosphate) or a metal borate.

19. A powder coating composition as claimed in claim 18, wherein the corrosion-inhibiting additive comprises zinc phosphate (preferably in  
15 spheroidal form) modified with zinc molybdate and rendered organophilic by suitable surface treatment.

20. A powder coating composition as claimed in claim 18, wherein the corrosion-inhibiting additive comprises dicalcium phosphate dihydrate.

21. A powder coating composition as claimed in claim 18, wherein the  
20 corrosion-inhibiting additive comprises an additive from any of classes (i) to (xi) as hereinbefore defined.

22. A powder coating composition as claimed in any one of claims 1 to 21, wherein the proportion of corrosion-inhibiting additive(s) incorporated before and/or during homogenisation of the composition is in the range of  
25 from 0.5 to 50% by weight, based on the total weight of the composition, for example at least 1%, 5% or 10% by weight and not exceeding 20%, 30% or 40% by weight.

23. A powder coating composition as claimed in any one of claims 1 to 21, wherein the proportion of corrosion-inhibiting additive(s) incorporated by dry-blending is no more than 7.5% by weight, preferably no more than 5% by weight.
- 5 24. A powder coating composition as claimed in any one of claims 1 to 23, wherein the total content of metallic pigment(s) and/or other non-film-forming additive(s) incorporated by dry blending does not exceed 10% by weight, based on the weight of the composition without the pigment(s) and additive(s).
- 10 25. A powder coating composition as claimed in any one of claims 1 to 24, wherein the particle size of the or each corrosion-inhibiting additive or component thereof is up to 25 microns, preferably no more than 10 microns, more especially from 2.5 to 7.5 microns.
26. A powder coating composition as claimed in any one of claims 1 to 15 25, wherein the particle size of any zinc oxide included in the corrosion inhibiting additive is in the range of from 0.1 to 10 microns.
27. A powder coating composition as claimed in any one of claims 1 to 26, which is a thermosetting system.
28. A powder coating composition as claimed in claim 27, which 20 incorporates a film-forming polymer selected from carboxy-functional polyester-resins, hydroxy-functional polyester resins, epoxy resins, and functional acrylic resins.
29. A powder coating composition as claimed in any one of claims 1 to 28, wherein the metallic pigment is a coated material.
- 25 30. A powder coating composition as claimed in claim 29, wherein the coating comprises silica or other inert inorganic material.
31. A powder coating composition as claimed in claim 29, wherein the coating comprises a plastics material.

32. A powder coating composition as claimed in claim 29, wherein the metallic pigment is coated with a colouring agent.

33. A powder coating composition as claimed in any one of claims 1 to 28, wherein the metallic pigment is carried in a polymer or plasticiser which is  
5 compatible with the film-forming polymer.

34. A process for forming a coating on a substrate, in which a composition as claimed in any one of claims 1 to 33 is applied to the substrate by a powder coating process resulting in particles of the composition adhering to the substrate, and forming the adherent particles into a continuous coating  
10 over at least part of the substrate.

35. A coated substrate obtained by a process as claimed in claim 34.

36. A coated substrate as claimed in claim 35, wherein the substrate is a metal substrate.

37. A coated substrate as claimed in claim 35, which comprises a non-  
15 metallic material.

38. A powder coating composition as claimed in claim 37, which comprises a plastics material, wood, a wood-based product, glass, glass fibre or a composite, ceramic or textile material.

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ABSTRACT

POWDER COATING COMPOSITIONS

5 Powder coating compositions comprise a film-forming polymer, a pigment providing a metallic effect, and a corrosion-inhibiting additive which serves to stabilise the pigment against attack caused by atmospheric humidity.

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